SYNOPSIS

Modification and Fabrication of Selected Natural Fibres into Thermal Bonded Structures for Hybrid Polymer Composites

By

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CONTENT

Chapter	Contents
I	Introduction
	Objectives of the study
II	Review of literature
III	Methodology
IV	Results of the study
V	Conclusions
	Recommendations

Introduction

Natural fibres play an essential role in rising "green" economy as their manufacturing process is energy efficient, renewable and also reduce carbon release. Increased environmental awareness, societal interest, new environmental rules and unsustainable petroleum consumption prompted consideration of environment friendly materials. The natural fibers are classified as bast fibers namely flax, jute, hemp, kenaf and ramie; leaf fibers such as agave, abaca, and pineapple; fruit and seed fibers such as coir, cotton and kapok, core fibers such as kenaf, hemp and jute, grass and reed fibers namely bamboo, elephant grass, wheat, corn and rice and other fibers from wood and roots. .*Agave americana* commonly known as American aloe or maguey belongs to monocotyledonous family called *Agavaceae* that gives leaf fibres which are coarse and hard. *Abutilon indicum* is a medicinal plant that belongs to the family '*Malvaceae'*, commonly known as Thuthi/Atibala. that yields bast fibres. Areca nut fiber belongs to the species *Areca Catechu* Linnaeusunder the family *Palmacea* gives fruit fibres.

The fibres as raw materials are directly converted into nonwovens and these are engineered fabrics which would satisfy the needs of technical textiles for interiors in mobile tech and built tech (K Savitha *et al.,* 2021). When two fiber components are brought together, each will contribute characteristics that are advantageous and less advantageous for the purpose. These individual characteristics exert a greater or smaller influence depending upon the blend properties of the components (Klein 2010). So, fibres are blended to obtain novel materials for specific end uses. Hybrid composites are the compound substances that are fabricated by combining two or more different kinds of fibres within a same matrix. Hybrid composites also find their application in aerospace, automobile construction and marine. The positive attribute of one kind of fibre could complement the lacking properties in other forms of constituents within the hybrid composites and this leads to the development of better performance and cost-effective products.

The composites made from natural fibres are advantageous as they are renewable resources and need low energy during their manufacturing process. These are not only biodegradable and renewable but also are advantageous because they are lightweight, economic, high strength and safe (Asma *et al.*, 2020). Natural fibres are extremely polar due to the presence of hydroxyl groups, which can easily establish hydrogen bonds with interacting resin matrices. Lignin and waxy compounds coat the fibres, preventing the hydroxyl groups from interacting with polar matrices. So, plant fibres must be subjected to physical or chemical or biological treatment to change the surface and structure in order to produce reactive hydroxyl groups and make the surface rough for adhesion with polymeric materials (Ahmad R *et al.,* 2019). The composites made using bio materials from local resources is cost effective compared to other polymer composites available

today. This bio material reinforced polymer composites can open up a new frontier for the industrialization at rural sectors.

In recent years, a number of investigations have been made which prove that the worth of natural fibers against their synthetic counterparts such as glass and/or carbon be reinforced polymer composites. The classic variety is epoxy resin, manufactured through polymerization, used as a thermoset polymer for adhesives and composites. Epoxy resins can to be fabricated to yield a number of different products with varying properties and the principal benefit being lower shrinkage over unsaturated polyester resins superior mechanical properties, resistance to corrosive liquids and environments, superior electrical properties, good performance at elevated temperatures, good adhesion to substrates are also the major advantages of epoxy resins (Ray *et al.*, 2016).

Today biopolymer composites are used for a wide range of applications. This research work focusses on the manufacture of biomaterial reinforced composite structure by fibre surface modification and blending to prepare hybrid materials. Considering the above facts, the study entitled "**Modification and Fabrication of Selected Natural Fibres into Thermal Bonded Structures for Hybrid Polymer Composites"** has been undertaken to make materials which would be suitable for application as building material in future with the following objectives.

Objectives of the study

The present study comprises of the following objectives,

- To understand the necessity of natural fibre building materials over the existing ones and select suitable natural fibres based on local availability for bio building materials
- To select suitable surface modification treatments to improve the functionality of natural fibres for preparation of compatible composite materials
- To fabricate and evaluate thermal bonded structure utilizing the surface modified natural fibres by blending
- To develop hybrid composite materials utilizing the prepared thermal bonded structure
- To evaluate the characteristics of hybrid composite materials

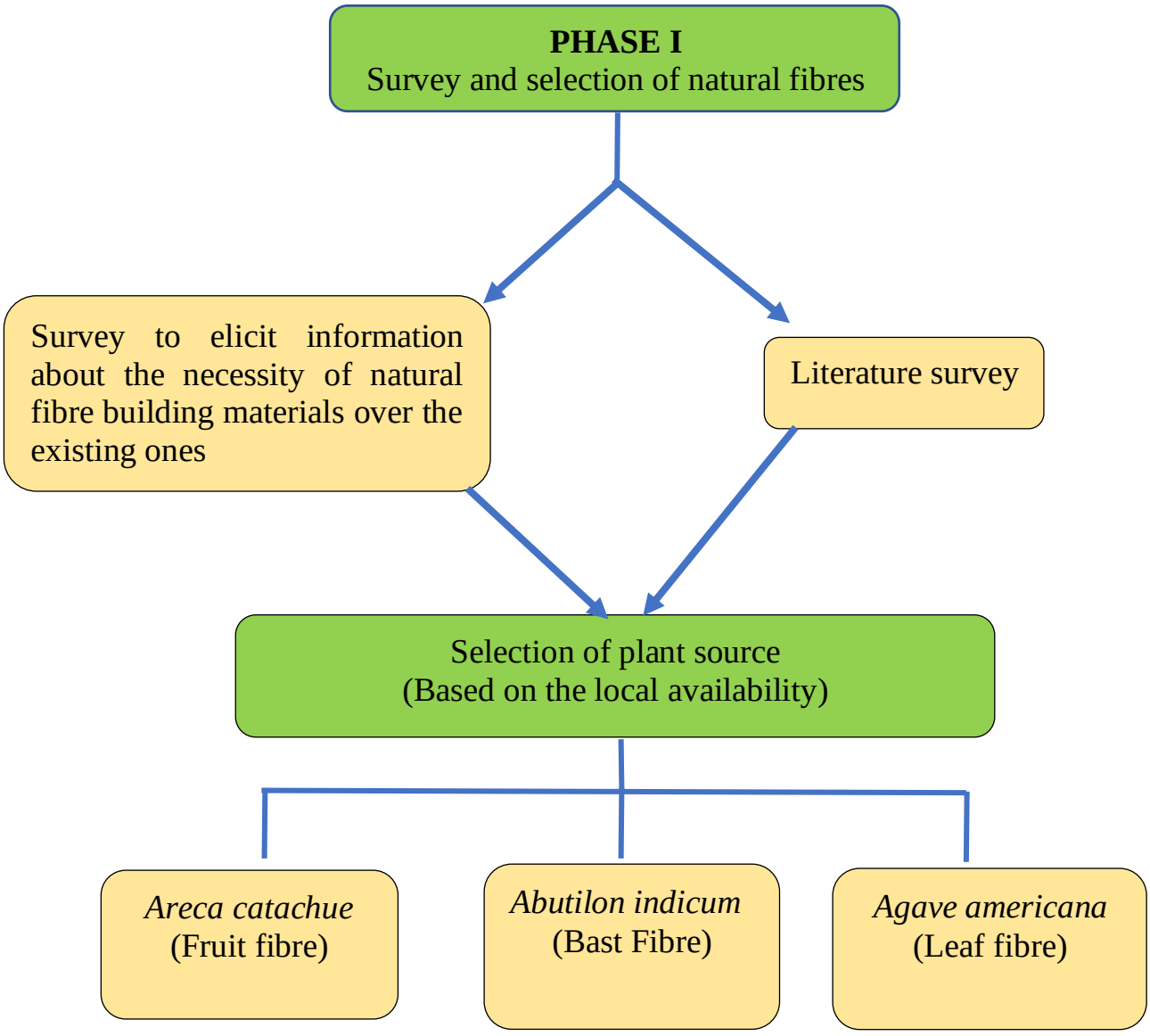
Review of literature

The literature survey carried out for the study are presented under the following topics.

- Textile natural Fibres and their classification
- Surface modification methods of natural Fibres
- Nonwoven techniques for natural fibres
- Role of natural fibres as reinforcement in composite preparation
- Importance and application of hybrid composites
- Potentiality of eco-friendly bio material reinforced hybrid polymer composites

METHODOLOGY

The methodology followed for the study comprises of four phases which is presented in flow chart (Fig. 1)



PHASE II
Fibre surface modification

Chemical treatments
- Alkalization
- Benzoylation
- Acetylation

Enzyme Treatments
- Cellulase
- Pectinase
- Cellulase + Pectinase

Nomenclature of Fibre Samples		
Untreated *Abutilon indicum* – **UNAB**	Untreated *Agave americana* - **UNS**	Untreated *Areca catachue* - **UNA**
Alkali treated *Abutilon indicum*- **ABAK**	Alkali treated *A. americana* - **SAK**	Alkali treated *Areca catachue* -**AAK**
Acetylation treated *Abutilon indicum* - **ABACE**	Acetylation treated *A. americana* -**SACE**	Acetylation treated *Areca catachue* -**AACE**
Benzoylation treated *Abutilon indicum* - **ABB**	Benzoylation treated *A. americana* - **SB**	Benzoylation treated *Areca catachue* - **AB**
Cellulase treated *Abutilon indicum*- **ABC**	Cellulase treated *A. americana* **-SC**	Cellulase treated *Areca catachue* - **AC**
Pectinase treated *Abutilon indicum*-**ABP**	Pectinase treated *A. americana* **-SP**	Pectinase treated *Areca catachue* - **AP**
Cellulase and Pectinase treated *Abutilon indicum*- **ABCP**	Cellulase and Pectinase treated *A. americana* - **SCP**	Cellulase and Pectinase treated *Areca catachue* - **ACP**

Evaluation of untreated and treated fibres

- Visual evaluation
- Spectrophotometric analysis
- Tensile strength
- Chemical constituents
- Moisture content and regain
- Density
- Fourier Transform Infrared (FTIR)
- Scanning Electron Microscope (SEM)
- Thermogravimetric analysis (TGA)
- Water absorbency

Treated Fibre samples selected for further study
- ABAK, ABP, SB, SP, AAK and AP

PHASE III
Fabrication –Thermal bonding

Fibre Proportions and Nomenclature of Fabric Samples		
Untreated	**Chemical treated**	**Enzyme treated**
Areca catachue (25) +*Abutilon indicum* (25) +*A. americana* (25) +pet (25) - **UNAABS**	*Areca catachue* (25) +*Abutilon indicum* (25) +*A. americana* (25) +pet (25) - **CHAABS**	*Areca catachue* (25) +*Abutilon indicum* (25) +*A. americana* (25) +pet (25) - **ENAABS**
Areca catachue (40) +*Abutilon indicum* (40) + pet (20) - **UNAAB**	*Areca catachue* (40) +*Abutilon indicum* (40) + pet (20) - **CHAAB**	*Areca catachue* (40) +*Abutilon indicum* (40) + pet (20)- **ENAAB**
Areca catachue (40) + *A. americana* (40) + pet (20) - **UNAS**	*Areca catachue* (40) + *A. americana* (40) + pet (20) - **CHAS**	*Areca catachue* (40) + *A. americana* (40) + pet (20) - **ENAS**
Abutilon indicum (40) + *A. americana* (40) + pet (20) - **UNABS**	*Abutilon indicum* (40) + *A. americana* (40) + pet (20) - **CHABS**	*Abutilon indicum* (40) + *A. americana* (40) + pet (20) - **CHABS**

Evaluation of thermal bonded fabrics

- Fabric thickness
- Fabric Weight
- Tensile strength
- Abrasion
- Thermal conductivity
- Water absorption and Wettability test
- Fourier transform infrared (FTIR)
- Scanning Electron Microscope (SEM)

Nonwoven Fabric Samples Selected for Further Study

- CHAS, CHAAB, CHABS and ENAABS

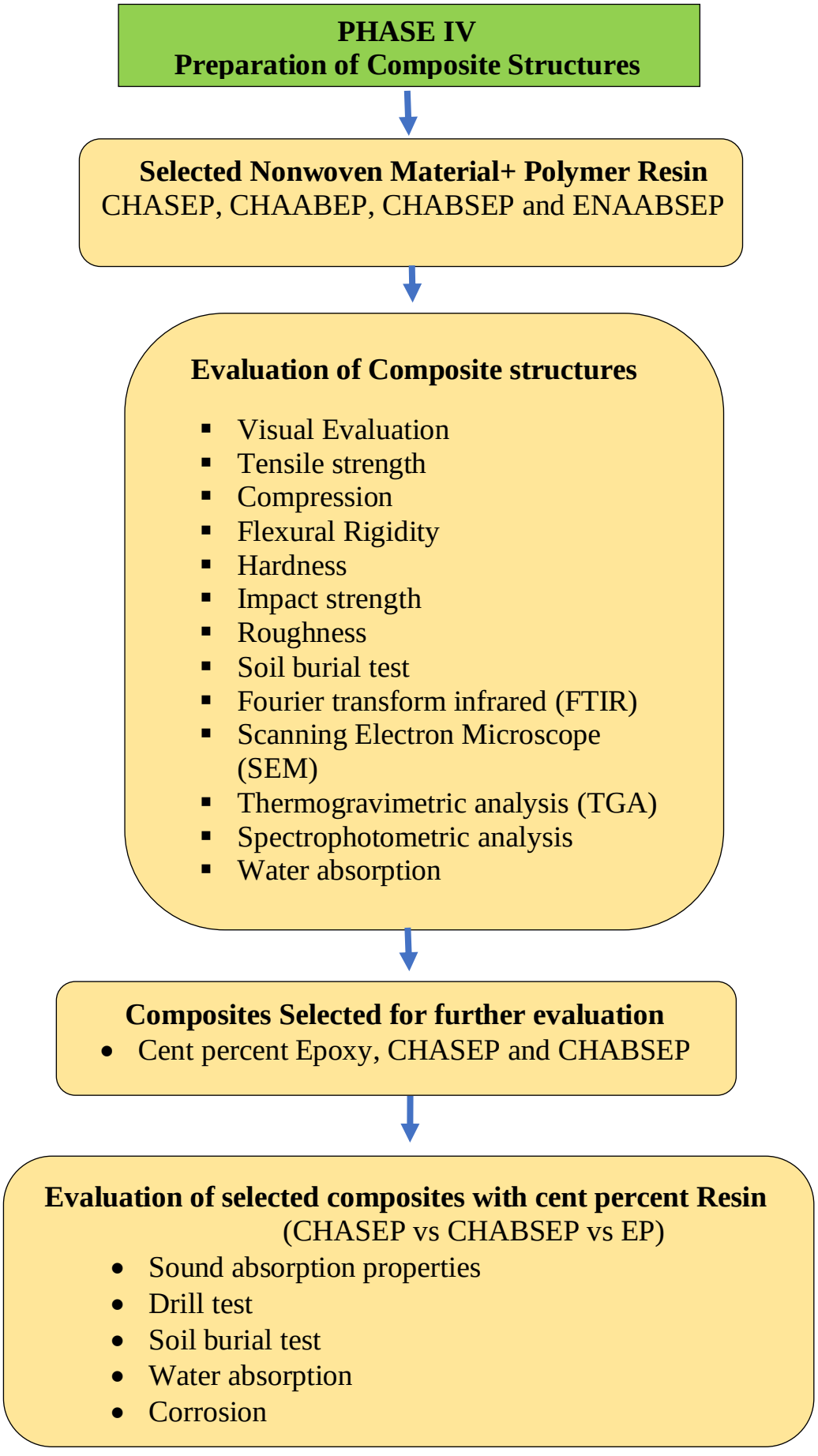


Fig. 1 Flowchart for methodology - all phases

METHODOOGY

The phases followed for carrying out the research work are vividly explained under the following heads. The nomenclature used for each sample and the samples selected after each stage of the study are clearly mentioned in the Flow chart. (Figure – 1).

PHASE I – Survey and selection of natural fibres

Survey was conducted among the persons involved in building construction for eliciting the information regarding the necessity for the use of building materials made for the natural fibres over the already existing materials using interview schedule as tool. As per the literature survey and local availability of the plants, namely *Areca catechu, Abutilon indicum and Agave americana* these plant sources were selected for fibre procurement. These plants were collected and submitted to Botanical Survey of India, Tamil Nadu Agriculture University, Coimbatore for authentication.

PHASE II – Fibre surface modification (21 Samples)

The selected plant fibres were subjected to chemical and biological treatments for surface modification to make it compatible for composite making. The chemical treatments carried out were Alkalization, Benzoylation and Acetylation. The biological treatments were done using the enzymes Cellulase, Pectinase and the combination of the two. The appropriate procedure was applied for each treatment and the fibres were evaluated for the essential properties. The treated and untreated fibres were assessed using various standard test methods for obtaining the salient properties which were compared and analysed. Then the fibres with favorable properties were selected for further study.

PHASE III - Fabrication – Needle punching and Thermal bonding (12 Samples)

The selected treated fibres were converted into thermal bonded materials in varying proportions using the recyclable fibre PET in minimum quantity. This was fabricated so, to obtain uniformity, strength, compatibility and good appearance while using as reinforcements for composite making. This thermal bonded material was then assessed using various standard test methods for obtaining the salient properties. The fabrics which gave favorable results were taken for further study.

PHASE IV – Preparation of Composite structure (4 Samples)

The composite structures were produced using the prepared thermal bonded materials as reinforcement and the polymer resin Epoxy along with Hardener as matrix individually. Also cent per cent Epoxy sample was prepared and used as control sample. Compression molding method was used to prepare the composite structures. Machining of the prepared composite structures were done as per standard dimensions to carry out the tests namely, Tensile strength, Compression, Flexural Rigidity, Hardness, Impact strength and water absorption. The samples which obtained the best

results were alone utilized for further evaluation. The selected samples were then subjected to special tests namely acoustic test, drill test, corrosion test, soil burial test and Liquid absorption tests.

Results of the Study

Phase I - Survey and selection of natural fibres

From the survey, it is obvious that high level of preference was expressed by respondents for eco-friendly products and already used fibre was coir in building application. The respondents were not much aware of other nonconventional fibers such as areca nut/sisal/abutilon fiber. It was understood from the literature survey and local geographical conditions that the plants namely *Areca catachue, Abutilon indicum* and *Agave americana* are available abundantly. Hence the above plants were selected for the fibres.

PHASE II – Fibre surface modification (21 Samples)

Chemical constituents and physical properties of fibres

The **lignin content** of the sample UNAB was noted to be reduced in the samples ABAK and ABB, but an increase was noted in sample ABACE. The **wax content** of the sample UNAB was reduced in samples ABB and ABAK, whereas a slight increase was noted in sample ABACE. The **moisture regain** of the fbre sample UNAB reduced in sample ABAK and drastically increased in samples ABB and ABACE. This decrease in moisture regain by alkylation process may be due to the increase in the hydrophobic property of the fibre. But by acetylation and benzoylation the hydrophilic property has been induced to the A. indicum fibre samples. **Moisture content** of the sample UNAB reduced in samples ABAK, ABACE ABB The **density** of samples UNAB was observed to be decreased in samples ABB and ABACE no change in density was noted in sample. Hence the maximum de lignification was observed in sample ABB. The wax content reduced in sample ABAK and ash content substantially reduced in sample ABACE. As far as moisture content is concerned, a substantial reduction was noted in sample ABB. Density was observed to have a substantial decrease in sample ABB. The statistical study of one-way ANOVA performed for the interaction between the chemical treated and untreated samples for cellulose and lignin contents showed a significance difference at 1% level. Benzoylation reduces the hydrophobicity of the fibre. which is also proved in this study as the sample ABB exhibits higher water absorption than sample UNAB. Acetylation modifies the surface of natural fibers making it more hydrophobic than untreated as the chemical reacts with the hydroxyl groups (OH) of the fibre. which is exhibited in the sample ABACE. The **water absorption** property increased drastically in sample SB. In areca nut husk fibres there was reduction in all the chemical treated samples of whichit was the maximum in the sample AB followed by the samples AAK and AACE. Among the enzyme treated samples the reduction was observed in

samples ACP (222.59 per cent) and AC (149.16 per cent). But an increase was observed in the sample AP (345.82 per cent).

Scanning Electron Microscopic Analysis

The morphological analysis by SEM study reveals that there is a significant change on the surface of fibres after treatments. The fibre is held together by the substances such as lignin and pectin which is well observed in the treated samples. On various chemical treatments, the fibres are noted to have splits on the surface in most of the cases which may be due to the roughness formed on the surface of the fibres. The removal of lignin and celluloses from the surface of fibres may be the cause of this morphological alteration.

PHASE III - Fabrication – Needle punching and Thermal bonding (12 Samples)

Physical properties of nonwoven fabric samples

- The **thickness and weight** of nonwoven structures prepared from the treated fibre exhibited more thickness than the untreated fibre utilized fabric samples
- The treated fibre utilized thermal bonded structures exhibited more **strength** in sample CHAS followed by CHABS CHAABS and ENABS than the untreated fibre utilized Thermal bonded fabric samples.
- The **tensile** strength and **bursting** strength analyses showed similar trend. that the sample CHAS was the strongest among all the nonwoven samples. The tensile strength when compared between the respective sets of samples, the sample ENAAB exhibited the highest strength among the samples UNAAB/ENAAB/CHAAB; the sample CHABS showed the highest strength among the samples UNABS/ENABS/CHABS and among the samples UNAABS/ENAABS/CHAABS the sample CHAABS showed the highest strength.
- Among the treated samples the **thermal resistance** was the highest in sample CHABS followed by sample ENABS and CHAS
- The **spray test** showed that the nonwoven samples had absorbency rate of 70 depicting partial wetting of the fabric surface
- The **SEM** appearance of nonwoven fabric sample UNAS exhibited the interlocking of the fibres. The entanglement is very high as per the SEM view in the sample ENAS which has improved the compactness and in turn the thickness which may be the reason for the increase in acoustic property. This same trend was observed in the comparison made between the samples UNABS, CHABS and ENABS. Due to better interlocking of fibres the thickness also was higher in both the treated fibre nonwoven structures compared with the untreated fibre interlocked sample. Among

the samples UNAAB, CHAAB and ENAAB the sample ENAAB showed highest interlocking. This may be due to the softness and flexibility gained while doing further fabrication process.

- Among the thermal bonded fabric structure, the **thermal degradation** occurred at three stages in untreated Areca Sisal fabric with maximum temperature Whereas Enzyme treated fabrics reached two stages of degradation. Among Areca *Abutilon indicum* fabrics untreated fabrics had three stages of thermal degradation followed by Chemical treated samples had two stages of degradation with minimum weight loss. Chemical treated *Abutilon indicum* sisal fabrics chemical treated fabrics decomposed at third stage. Among Areca *Abutilon indicum* Sisal fabrics chemical treated fabrics degraded at third stage. The thermal stability was the highest in chemical treated Areca sisal blended fabric structure. The thermal stability was the highest in chemical treated Areca sisal blended fabric structure.
- The acoustic properties when compared between the respective sets of samples, the sample ENAS showed the maximum Sound Absorption Coefficient (SAC) among the samples UNAS/ENAS/CHAS; the sample ENAAB showed the maximum SAC values among the samples UNAAB/ENAAB/CHAAB; the sample CHABS showed the maximum SAS among the samples UNABS/ENABS/CHABS, and among the samples UNAABS/ENAABS/CHAABS the sample CHAABS showed the highest SAC values.

Based on the essential properties studied for the prepared nonwoven samples, the samples which gave as the favorable results were considered for further study.

PHASE IV – Preparation of Composite structure (4 Samples)

Physical properties of composite Structures

All the tests showed better results in the fibre reinforced hybrid polymer composites than the composite prepared from cent per cent epoxy resin.

- Among composite structures the Tensile strength was found to be highest in the sample CHABSEP followed by the sample CHASEP. The reason for the highest Tensile strength in the samples may be due to the inherent properties of the abutilon fibres over araca nut husk fibre. This may also be due to the best compatibility between the reinforcement and matrix and absence of voids.
- The composite sample CHASEP show the maximum flexural strength followed by sample CHABSEP
- Compression strength was the maximum in sample CHAABEP followed by

CHASEP. This may be due to the ability of the fibres to absorb stress transferred from the matrix and this enhancement is due to the interface bonding between the reinforcement and matrix

- The sample ENAABSEP shows the highest impact value among the composite samples
- The hardness shows the ability of a material to resist plastic deformation under indentation to be determined. This enhancement in hardness is due to the uniform distribution of the load on the fibres
- The absorbency of the sample noted to be medium in sample CHABSEP after 24hrs and 48hrs whereas the sample CHASEP showed lesser absorbency rate than CHABSEP. The leastabsorbency rate was noted in the sample CHAABEP and no absorbency was noted in cent percent epoxy sample.
- SEM appearance of composites structures show the compatibly of resin and reinforcement
- All the composite materials exhibited three stages of thermal degradation. The thermal stability was the highest in sample CHASEP with more than 100 percent degradation followed by sample CHABS, CHAABS and ENAABSEP
- The samples show slight fibre pulling in the drill test carried out at various drill bits.
- The SAC of samples CHABSEP and CHASEP showed raise in Sound Absorption Coefficient between 400 and 500 frequency bend. This shows a gradual raise beyond 0.2 SAC between 1800- 2100 frequency. It has a tendency to move higher depicting higher acoustic property. The transmission loss of the samples CHABSEP and CHASEP was observed between 800 to 1600 frequency band, higher transmission less above 0.15db was noted which slightly declined and again a raise was observed beyond 1800hz. Beyond 2400hz the sample CHASP exhibited higher transmission loss as five peaks were observed in the sample. Higher the transmission loss, lower the sound passes through the composite panel. The higher transmission loss was noted in sample CHASEP which shows that it can reduce the noise when applied.

Conclusion

Plant-based natural fibers have a great advantage over synthetic fiber because of their lightweight, nonabrasive, nontoxic, and biodegradable nature. The selected natural fibres obtained from the plants sources namely *Areca catechu, Abutilon indicum and Agave americana* could be well modified to make it suitable for composite manufacturing as it had improved the compatibility. The fibre blending and fabrication of these fibres into thermal bonded structure is possible with goo interlocking of fibres. The hybrid polymer

composite structures could be easily prepared with good compatibility between the natural fibre reinforcemtns and matrix. The natural fibre reinforced polymer composites are cost effective when compared with the commercial boards. These boards can be used as acoustic materials, panels and many more products fulfilling functional and aesthetic aspects for building interiors as there is a need for ecofriendly building materials. Based on the properties required to satisfy the end uses, these novel materials may be utilized.

Recommendations for further study

- The natural fibre reinforced polymer composites structures can be produced as large panels for building interior applications
- The natural fibres can be modified with different other treatments which can enhance the properties further.
- The reinforcement matrix proportion can be altered and analysed for results.

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